

# Vapor Performance Testing of Filter Materials and Filter Canisters



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## Background

### Introduction

Filters protect the first responder and warfighter from toxic vapors. It is necessary to test filters with toxic vapor to prove that deployed filters will protect personnel. Results must be reliable to human toxic effects and to realistic threats.

Collective protection equipment, to include gas masks, cannot be field-tested with toxic chemical agents against human participants. Agent field test performance has been predicted by combining simulant field test data with the results of laboratory component tests using toxic agents.

To address this shortcoming, DPG developed the Swatch Including Filter Test (SWIFT), a modular near real-time permeation apparatus [1]. Liquid and vapor permeation and off-gassing of filter materials and components can be measured. Simulants or chemical warfare agents (CWAs) may be disseminated in either vapor or liquid states. Pliable and rigid materials as well as small filters may be tested. Only filter testing with vapor is discussed here.

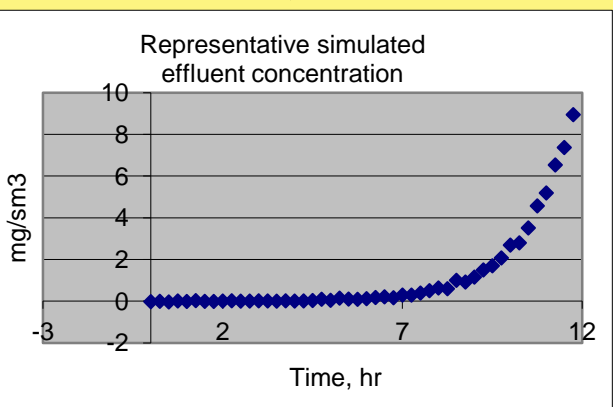
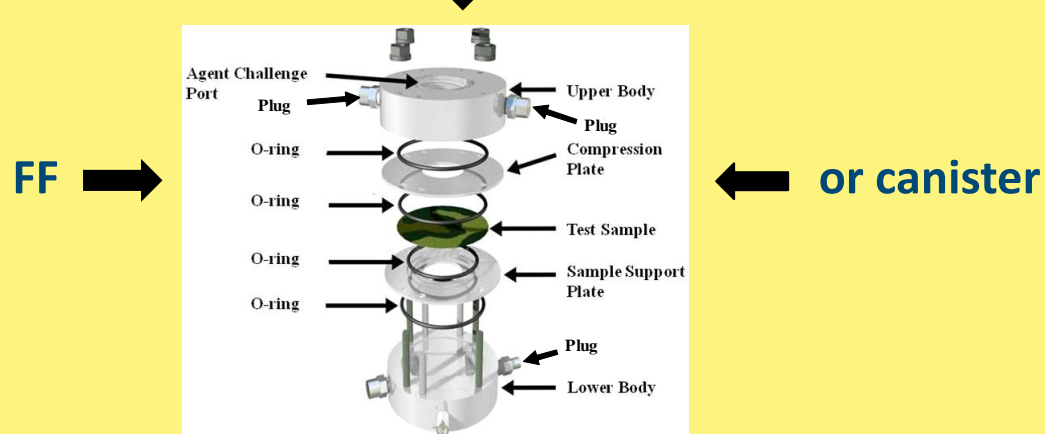
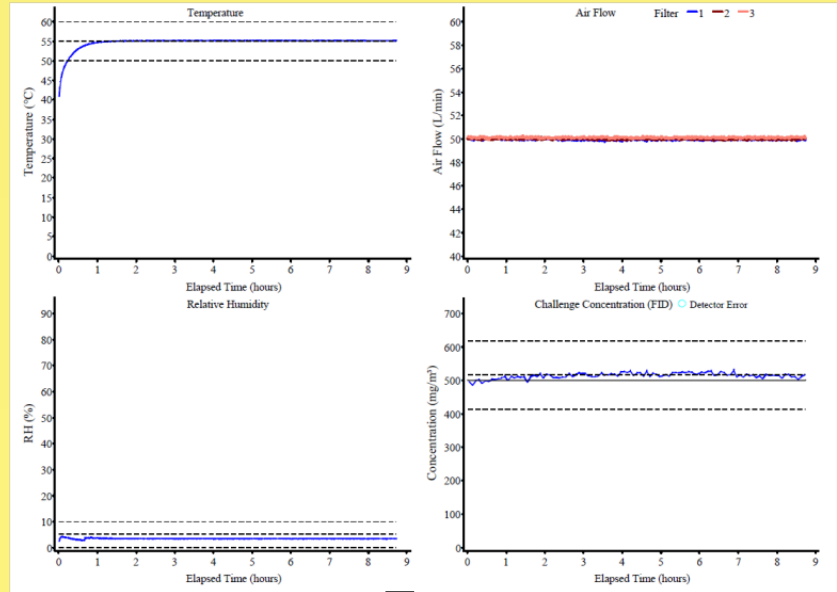
### Approach

Three test items were placed in a thermostat-controlled enclosure that fit most chemical fume hoods. Temperature, relative humidity, and challenge vapor concentration were controlled. Challenge and effluent vapor concentrations were measured every five minutes. Other values were recorded every minute.

A standard filter canister used in military protective masks was tested as an example of a small-scale filter, as well as swatches of filtration fabric (FF). The FF is designed to remove CWA and biological warfare agents from incoming air, while allowing exchange of breathable gases.

Example data are shown, but do not necessarily predict how the system would respond to an actual threat.

### Challenge Conditions

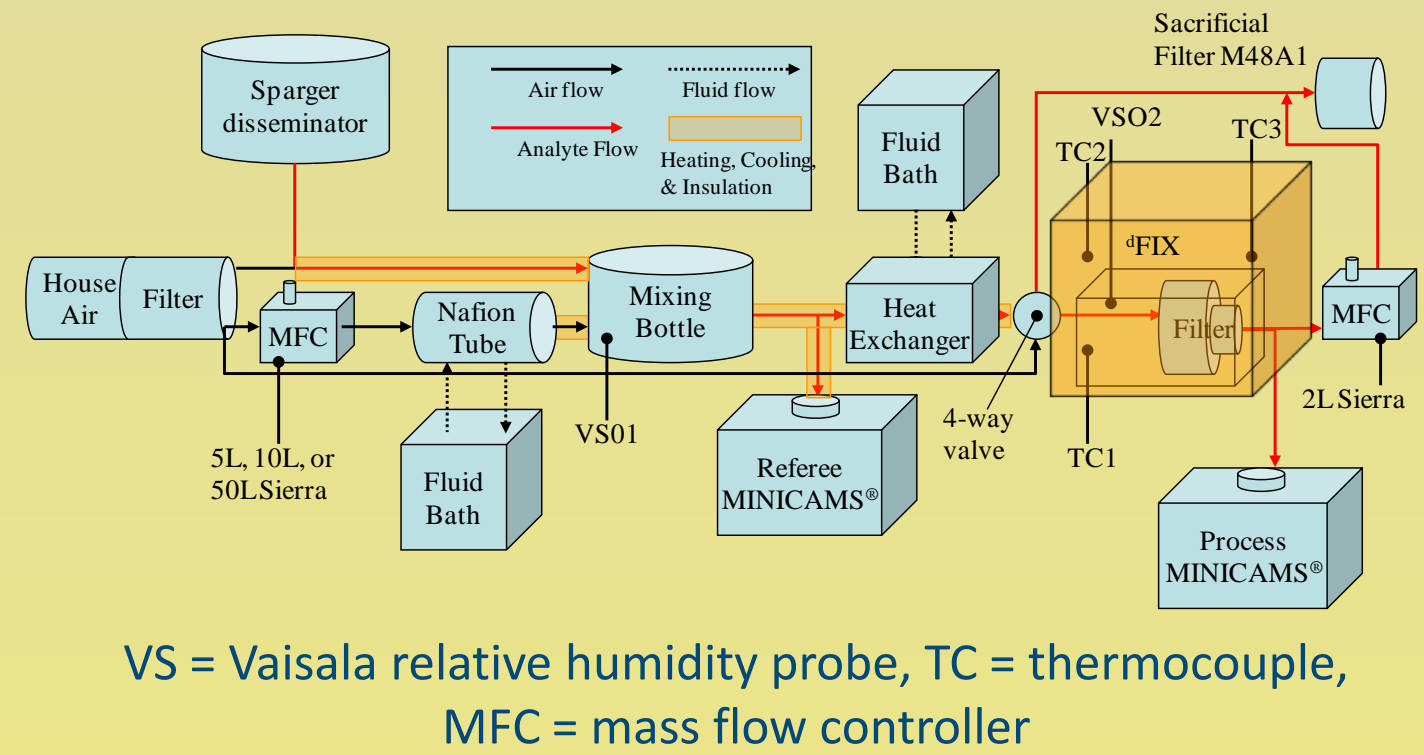


## Procedures and Conditions

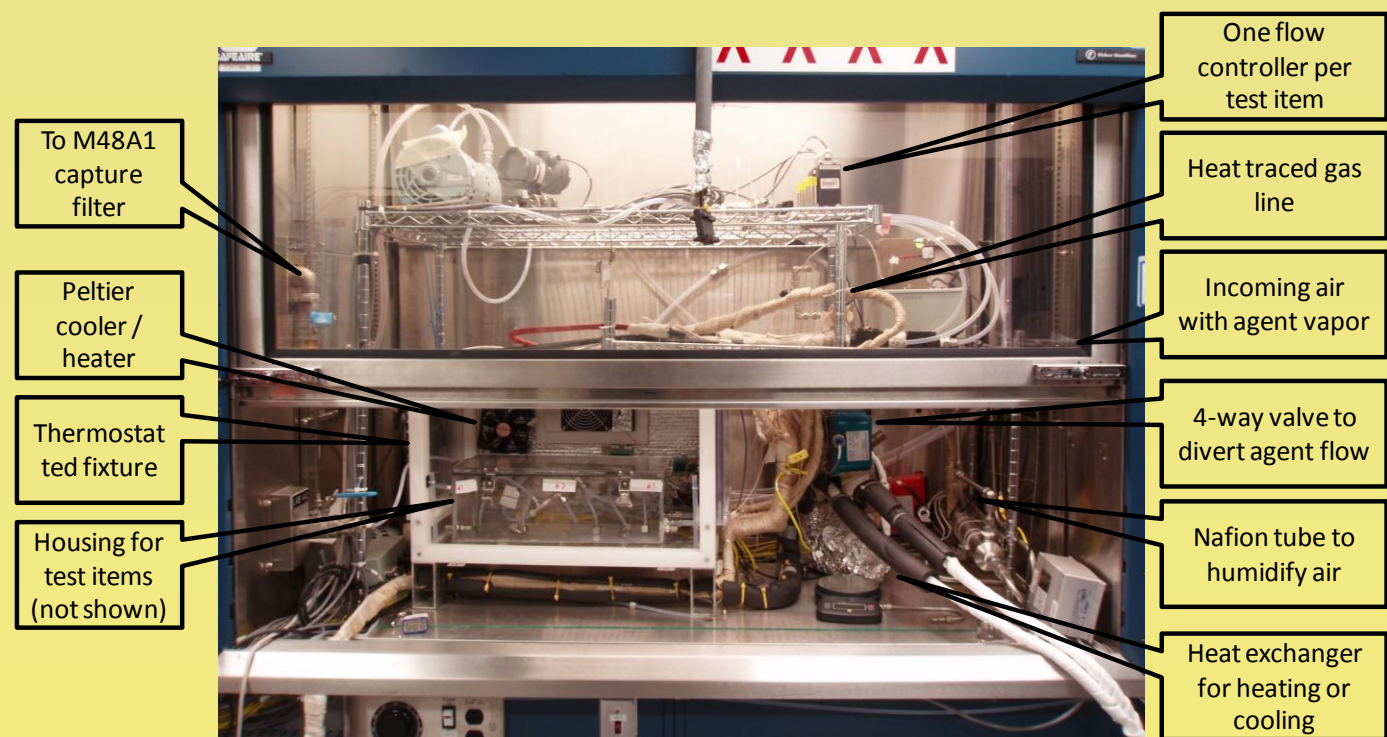
- Before vapor challenge, some test items were exposed to battlefield contaminants (BFCs) in a shipping container. BFCs were chosen by a multi-service group of experts as most likely to be encountered on the battlefield and to impair performance. They included either a mist of fog oil battlefield obscurant, or diluted exhaust from a JP-8 fueled generator, and were drawn through test items at ambient pressure.
- JP-8 exhaust exposure was 72 h. 54 mL of fog oil was disseminated for three 1 h periods. FF was exposed at a differential pressure ( $\Delta P$ ) of 0.025 inches water gauge (iwg). Exposure flow rate was 50 standard liters (SL) / min per filter canister.
- In the laboratory, FF from the test items was die-cut into 2" coupons. Each coupon was sealed into a standard swatch test cup, milled deeper to hold the coupon. Differential pressure ( $\Delta P$ ) was controlled across each coupon for each trial;  $\Delta P$  varied from 0.02 to 0.10 iwg. The air flow through each coupon ranged from about 0.1 to 1.0 standard L/min (SL/min). Standard flow was corrected to 21.1°C, 1 atmosphere barometric pressure.
- Air flow through each filter canister was 50 SL/min. Each canister was challenged at a much faster flow rate than a FF sample because it held much more adsorbent.
- Filters were equilibrated at test temperature in a dry air flow before vapor challenge.
- Challenge agents: sarin (GB), soman (GD), and VX. A different simulant was used for each agent. Filter canisters were not tested with VX or its simulant.
- Conditions: ambient pressure. Temperature 5°C to 55°C. Relative humidity (RH) at high temperature: 0 to 85%, at low temperature: 0 to 65-70%, measured and not inferred. Vapor concentration 1 to 5000 mg/sm<sup>3</sup> depending on compound, temperature, and RH. Not every combination of conditions was tested. A D-optimal design of experiments was used to select the combination of challenge conditions.
- Effluent concentration through each filter was measured using a miniature automatic continuous air-monitoring system (MINICAMS). Each MINICAMS® consisted of a preconcentrator, gas chromatograph, and flame ionization and photometric detectors (FID, FPD). Calibration range was 0.000833 to 15 mg/sm<sup>3</sup>. Trial was ended when effluent reached 10 mg/sm<sup>3</sup> or after 48 h (for the canister) or 12 h (for the FF).
- Challenge concentration was measured with a MINICAMS® with an attached low-volume sampler. Calibration range: 100 to 1500 mg/sm<sup>3</sup>.
- The mass gained by each filter was measured.

## Fixture, Results, and Analysis

### Schematic and Photograph for Small Canister Filters



VS = Vaisala relative humidity probe, TC = thermocouple, MFC = mass flow controller



Trials were performed in two SWIFT fixtures of similar design. Environmental conditions were measured at more than ten points throughout the fixture. Surfaces exposed to gas were stainless steel. Lines were heat-traced so that CWA vapor would not condense on a cold surface. Safety interlocks and safe procedures were used to test with large amounts of CWA. Waste vapor was captured in a large filter.

For FF testing, the sparger was replaced by a syringe pump to meter a low flow rate of liquid agent into a heated tee.

### Data

#### Challenge Conditions and Vapor Effluent

Results passed Quality Control (QC) measures. At the same test conditions, most results were repeatable day to day and fixture to fixture. These standards were used to accept results from each trial, but not to accept the test item:

- Over 90% of temperature readings within  $\pm 5^\circ\text{C}$  of target.
- Over 90% of the RH readings within  $\pm 5$  percent RH of target.
- Mean challenge concentration within 20 percent of target and over 90% of concentration measurements within 20 percent of mean.
- The challenge Ct must equal or exceed the minimum challenge Ct: GB & its simulant 2573 mg min/sm<sup>3</sup>, GD & its simulant 2518, VX and its simulant 431. Ct is the challenge concentration integrated over time during the trial.

#### Flow Rate through FF

The flow rate through a FF swatch depended linearly on the  $\Delta P$  across the swatch: flow rate Q (SL/min)  $\approx 9.1 * \Delta P$  (iwg).

#### Mass Gain

Trends for mass gain were determined for each challenge compound, for filter canisters and FF.

### Analysis of FF Effluent Concentration

#### 1: Developed Mathematical Models of Concentration

Considered filtration physics. Three models were used: filtration, inefficiency, or full. The filtration model was that of Gerry Wood [2], simplified to an exponential:  $C = A \exp(K_f t)$ . C was effluent concentration, t was elapsed time, and the parameters were varied to fit data. The leakage or inefficiency model described vapor leaking between the filter particles:  $C = B [1 - \exp(-K_2 t)]$ ; the parameters were varied to fit the data. The full model was the sum of the filtration and leakage models.

#### 2: Data Preparation

Filters were challenged with vapor in groups of three. Data from each filter was treated independently. Only customer-accepted data were analyzed. Points with detector errors were removed. Data from FPD and FID detectors were merged.

#### 3: Background Correction

Data were reviewed. A method was selected to mathematically correct C for the background concentration of vapor remaining from the previous trial. Data points outside the detector calibration range were removed.

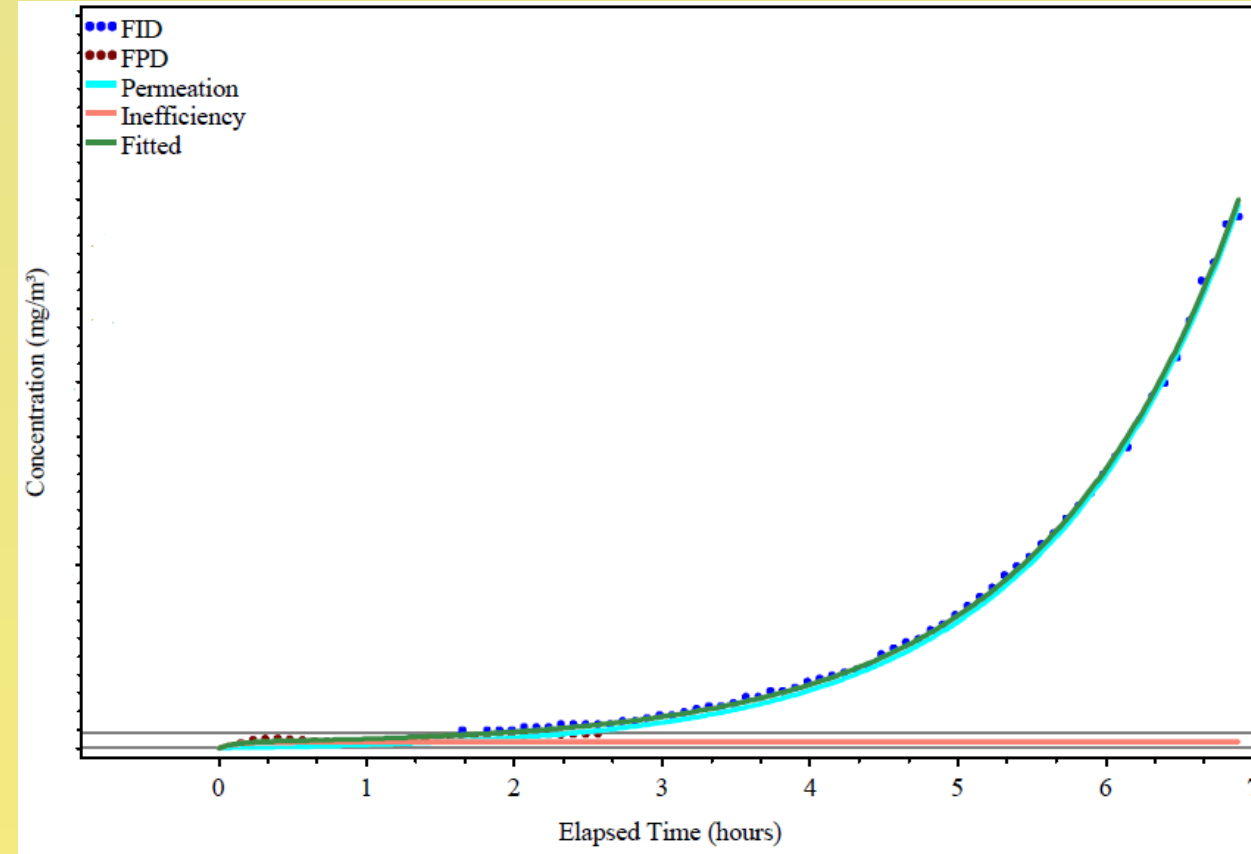
## Data Analysis, continued

#### 4: Time to Reach Military Exposure Guidelines (MEGs)

Each background-corrected data curve was assessed against the MEG concentration for the agent [3]. The agent MEG was used to assess data for the corresponding simulant. MEG values were taken from military toxicology guidelines. There were separate MEG values for each agent. There were separate values for 10 minutes, 1 hour, 8 hours, and 24 hours. From each curve a breakthrough time was derived, which was the time for C to exceed the MEG.

#### 5: Fitting to Model

Each data curve that was background-corrected in step 3 was fitted to one of the models developed in step 1.



Full model fitted to data from a trial

Good fits were obtained from about 0.0008 to 3 mg/sm<sup>3</sup>, a range of approximately 4000. 54 sets were fitted by full model combining permeation and inefficiency, 10 by inefficiency, and 84 by permeation model.

#### 6: Predicting C for One Trial

Fit parameters were used to predict C for the conditions of this trial. C was predicted every 30 minutes till end of trial.

#### 7: Predicting C at Any Condition

Predicted Cs were interpolated, to determine C at ANY condition or time in the range tested. Equation:

$$\ln C = m_0 + m_1 + m_2 C_0 + m_3 \Delta P$$

Where  $m_0$  = the intercept for simulant,  $m_1$  = a constant that is zero for simulant and non-zero for agent,  $m_2$  = slope of the trend with challenge concentration (sm<sup>2</sup>/mg),  $C_0$  = challenge concentration (mg/sm<sup>3</sup>), and  $m_3$  = slope of the trend with  $\Delta P$  (iwg<sup>-1</sup>). Values of the parameter in the Equation were calculated at nine times over the duration of the trial.

#### 8: Agent-Simulant Relationship (ASR)

A theoretical framework is needed to predict NRT performance of filters in the laboratory. The model should also predict the performance of fielded filters.

As part of the model, an ASR is needed to predict component performance with agent from component performance with simulant. The ASR for a component such as a filter does not necessarily predict the ASR of the system.

For this work, the ASR is defined as the ratio of estimated agent C to estimated simulant C. The GB-simulant and GD-simulant ASRs were calculated at nine times over the duration of the trial.

## Summary

### Filter Materials

About 400 trials were performed on 1200 test items. Both small canisters and FF held off vapor till media were saturated and vapor broke through. After breakthrough, effluent concentration rose exponentially with time, typically doubling in one to ten hours.

The effect of flow rate, challenge concentration, prior BFC exposure, temperature, relative humidity on breakthrough time was quantified.

The GB simulant closely simulated GB breakthrough. The GD simulant closely simulated GD breakthrough. The VX simulant simulated VX breakthrough.

The necessary data have been gathered and reduced to mathematical equations for use in future predictions.

### Caveat

This test was not designed to directly simulate actual test conditions. Therefore, the performance of an actual fielded filter should not be directly deduced from these data.

### SWIFT Fixtures

The SWIFT system has proven to be a capable and versatile apparatus in which to test chemical and biological (CB) protective materials and equipment. To date, the SWIFT has been used for over 400 trials. The small-scale filter, filter material, permeation, and off-gassing configurations of the SWIFT have met most community and test-specific needs.

Within DoD, six programs have used SWIFT fixtures and have accepted the majority of test data. Operational test agencies have been part of each assessment and the data have met the needs of each test program. Test reports are available from the Joint Program Executive Office (JPEO) for completed tests.

The SWIFT fixture meets all current requirements except for the requirement to present a dynamic predetermined vapor challenge. SWIFT system configurations are in the process of accreditation.

### Future Work

Personal protective equipment (PPE) will continue to be tested at DPG. A SWIFT can be used to characterize vapor off-gassing before and after a test item has been decontaminated, to evaluate the efficacy of decontamination. A SWIFT can be adapted to challenge with different flow rates. In addition, toxic industrial chemicals (TICs) can readily be disseminated, using a 1-stage or a 2-stage dilution system if necessary.

### Acknowledgements

Personnel from the West Desert Test Center, Jacobs Dugway Team, IP Network Solutions, and Dugway Data Services Team coordinated and performed the test, processed trial data, and prepared test documents.

### References

- [1] Verification and Validation of Near-Real Time (NRT) Swatch Including Filter Test (SWIFT) System Configurations using the Dugway Fixture (dFIX): Swatch Permeation, Off-gassing, Adsorptive Media, and Small Scale Filters Filtration, draft, October 2012.
- [2] Gerry O. Wood, "Estimating Service Lives of Organic Vapor Cartridges," American Industrial Hygiene Association Journal, 55(1), 11-15, January 1994.
- [3] U.S. Army Center for Health Promotion and Preventative Medicine (USACHPPM), U.S. Army Aberdeen Proving Ground, Maryland, Health-Based Chemical Vapor Concentration Levels for Future Systems Acquisition and Development, USACHPPM 64-FF-0722-07, February 2008 (July 2008 Update).

### First Responder and Warfighter

Thank you for protecting the population.  
Testers test equipment that protects you!